

# LABNOTES

*Fall 1996*

The Newsletter of the Wisconsin Laboratory Certification and Registration Program  
Program Info: (608) 267-7633 Program Fax: (608) 267-5231

## CHANGES IN THE CERTIFICATION PROGRAM

### INTRODUCING: NEW REGIONAL AUDITORS!



Effective October 1, Brenda Howald assumed responsibility for municipal laboratories located in the Southern District.

Brenda has worked as a permit drafter at the Southern District for the past four years. Prior to coming to the DNR, she worked in both Quality Assurance and as the Inorganic Laboratory Supervisor at the former Warzyn's analytical laboratory. Her depth of experience in inorganic testing will make her an excellent addition to the regional audit team. Brenda is located at the Fitchburg District Office, (608) 275-3462.

Susan Scobell-Watson has been named the successor to Tom Atchison as the individual responsible for performing municipal laboratory evaluations in the current North Central District.

Susan is currently a Permit Coordinator, and will be adding this additional responsibility.

Her work with the permitting process gives her a familiarity with the facilities, providing a great complement to the auditing role. Susan is located at the Rhinelander District Office, (715) 365-8945.

We would like to welcome both of these new certification officers on board, and wish them the best of luck with their new assignments.

### In this issue...

Central Office Loses An Auditor .....	2
Nomination Forms For the 1997 Registered Lab of the Year Award Available .....	2
Bureau of Drinking Water & Groundwater Proposes Amendments to Chapter NR 140 .....	3
Laboratories Allowed Flexibility in Mercury Digestion .....	4
Focus on Analytical Techniques: Dry Purging .....	5
Oil and Grease - Hexane Isn't the Only Thing That's Different ...	6
Reference Sample News - Changes in the EPA Programs .....	7
Transferring Ownership & Maintaining Laboratory Certification .	8
News & Information Briefs .....	9
The Auditor's Corner .....	10
Facility Requirements for Landspreading Sludge .....	11

## ***Regional Responsibilities Will Change as a Result of the Reorganization***

The Department is moving forward with its Reorganization efforts, albeit on a delayed timeline. The anticipated implementation date for the switch from six Districts to five geographical Regions is early next year. While most laboratories will not notice a change in service, several municipal wastewater treatment facilities may be affected as Regional boundaries are shifted and their facility is assigned to a new staff member. The Laboratory Certification Program is investigating ways to continue to provide a high level of service to our small registered facilities throughout the Reorganization. It is likely that over the next two years many facilities will be assigned new auditors for Laboratory Certification. In the meantime, please continue to work with the same certification people that you have in the past.

## ***CENTRAL OFFICE LOSES AN AUDITOR***



Effective September 1st, the Laboratory Certification Program lost an auditor. Ms. Debra Piper, an auditor with the program since 1993, announced her resignation. Debra is best known to our biomonitoring laboratories as she visited all of them over the past several years. We will miss her good work and wish her well in her new position with a firm in the State of Illinois. We wonder if she will turn into a CHICAGO BEARS fan?

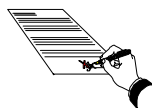
On a more serious note, the loss of an auditor stresses the program for an extended period of time. It will take several months to staff the position and then the training process begins. Training usually takes up to one year depending on the experience of the new person. In the meantime, we will do all that is possible to maintain a high level of service to laboratories participating in the program.

## ***NOMINATION FORMS FOR THE 1997 REGISTERED LAB OF THE YEAR AWARD AVAILABLE***



In 1996, the Laboratory Certification Program began the "Lab of the Year" award as a recognition program intended to reward two Wisconsin laboratories, one large and one small registered laboratory for their commitment to data quality. The awards are presented at the Natural Resources Board Meeting in March of each year. The DNR presented its first registered Lab of the Year awards to Modine Manufacturing and Bloomer WWTP in March of 1996. We received many qualified nominations for the awards, and selection of the two recipients was difficult. The first year of these awards was very exciting and successful, and we hope to continue this trend for 1997.

Nominations for 1997 are now being accepted, and are open to DNR staff as well as anyone in the public, but laboratories may not nominate themselves for this award. Again this year there are two award categories: one for small laboratories registered only in Categories 1 through 4 (BOD, CBOD, solids, ammonia, phosphorus, oil and grease, HEM) and one for larger laboratories that are certified in more test categories than just 1 through 4. Nominees must be currently registered with the Wisconsin Department of Natural Resources and must be physically located within the State of Wisconsin. Certified laboratories will not be considered.



To nominate a Wisconsin registered laboratory simply complete a nomination form available from the Wisconsin Department of Natural Resources, c/o Carol Lochner- SS/6, 101 S. Webster St., P.O. Box 7921, Madison WI 53707 or by phone from Carol at (608) 267-7633. The nomination forms are also available on the World Wide Web at {<http://www.dnr.state.wi.us/eq/lc/loyform.htm>}. Attach a brief summary no more than three pages long of why you think a particular laboratory deserves the award. Be sure you can clearly

justify, with specific examples, which of the following criteria you feel the nominee meets (the nominee does not necessarily have to meet all of the criteria to receive the award):

- ◆ How much effort has the facility made to correct non-compliance issues?
- ◆ Within the facility, are analysts free to make decisions that affect data use and do managers have a long term commitment to producing high quality data?
- ◆ Has the facility demonstrated the desire to comply with the Wisconsin Certification Program and gone beyond the normal expectations?
- ◆ Does the laboratory use its QA/QC data to evaluate data quality?

Nominations for the 1997 awards must be received by December 31, 1996 in order for the candidate to be considered. Award winners will be decided by a nomination committee consisting of DNR staff and Certification Standards Review Council members. Good Luck!

## ***BUREAU OF DRINKING WATER & GROUND WATER PROPOSES AMENDMENTS TO CHAPTER NR 140***

The Department of Natural Resources will be proposing new standards for ch. NR 140 (Groundwater Quality), Wis. Adm. Code, as requested by the Department of Health and Family Services. The Department will also propose some minor language amendments along with these new standards. The Department plans to request approval to hold public hearings on the changes at the January Natural Resources Board Meeting. The public hearing dates have not yet been determined, but will probably be scheduled in February or March of 1997. Please contact Steve Karklins in the Bureau of Drinking Water and Groundwater at (608) 266-5240 for more information. The following new standards will be proposed in s. NR 140.10 Table 1:

**TABLE 1.**

Parameter	ES	PAL	Parameter	ES	PAL
Acenaphthylene	5	0.5	Hydrogen sulfide	30	6
Anthracene	3	0.6	Methanol	5000	1000
Bentazon	300	60	N-Nitrosodiphenylamine	7	0.7
Benzo(b)fluoranthene	0.2	0.02	Prometon	150	30
Beta particle and Photon radioactivity	4 millirem/yr	0.4 millirem/yr	Pyrene	250	50
Boron	600	120	Pyridine	10	2
Carbon disulfide	1000	200	Radium 226 & 228	5 pCi/l	0.5 pCi/l
Chrysene	0.2	0.02	1,1,1,2-Tetrachloroethane	70	7
Cobalt	40	8	1,2,3-Trichloropropane	60	12
Dibutyl phthalate	100	20	1,2,4-Trimethylbenzene	50	10
Fluoranthene	400	100	1,3,5-Trimethylbenzene	50	10
Gross alpha particle radioactivity	15 pCi/l	1.5 pCi/l	Vanadium	30	6
N-Hexane	5000	1000			

\*\*All units in µg/l unless specified.

### ***Desk Reference Available***

Scheduled to coincide with the proposed amendments to Ch. NR 140, the Department of Natural Resources Bureau of Drinking Water and Groundwater has announced the availability of two newly created groundwater sampling documents titled *Groundwater Sampling Desk Reference*, PUBL-DG-037 1996, and *Groundwater Sampling Field Manual*, PUBL-DG-038 1996. These two new documents replace the 1987 Department documents (PUBL-WR-153 1987 and PUBL-WR-168 1987). Please recycle any copies of the older documents and help get the word out that everyone collecting groundwater samples and measurements should be using the new September 1996 versions.



In the past, these documents were provided to the public free of charge, but the Department can no longer afford to do this. Non-department staff must now purchase the documents through Document Sales (608) 266-3358 by requesting Stock #1278 to purchase the *Groundwater Sampling Desk Reference* and Stock #1729 to purchase the *Groundwater Sampling Field Manual*. The cost for both documents is around \$10. These documents will also be available on the DNR's World Wide Web site.

### ***LABORATORIES ALLOWED FLEXIBILITY IN MERCURY DIGESTION***

The Laboratory Certification Program has traditionally insisted on various directives for digesting samples to be analyzed for mercury. We are now allowing some flexibility in the physical conditions and chemicals used to accomplish digestions. This flexibility is based on options already available in various procedures approved for mercury analysis.

Aqueous samples can be digested by any of the following physical processes:

- ◆ Heating in a water bath for two hours at 95°C. This is the procedure recommended in most authoritative sources.
- ◆ Heating in an oven at 95°C for three hours. This procedure is allowed as an alternative to the water bath digestions in *Methods for Determination of Inorganic Substances in Water and Fluvial Sediments* of the US Geological Service, but it is NOT approved for drinking water.
- ◆ Heating at 105°C on line in an automatic analyzer. This is the procedure described in Method 245.2, but it may not be suitable for all aqueous matrices: chlorine, volatile organic materials, and solids interfere. Experience shows that the procedure works well for clean samples.
- ◆ Heating in a boiling water bath for two hours. This procedure is also allowed by the US Geological Survey Manual, but it may cause poor recoveries.

Solids or semi-solid samples can be digested by any of the following physical processes:

- ◆ Heating in a water bath at 95°C, first for two minutes, and then for 30 minutes, after adding oxidant.
- ◆ Heating under pressure in an autoclave at 121°C and 15 psi for 15 minutes.

Aqueous samples are digested using a combination of sulfuric and nitric acids, potassium permanganate, and potassium persulfate.

Solids and semi-solid samples are usually digested using a combination of hydrochloric and nitric acid (aqua regia) and potassium permanganate. This is traditionally the cocktail added when a water bath is used. The following are also acceptable:

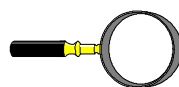
- ◆ A combination of sulfuric and nitric acids with potassium permanganate. This is typically the mixture used with the autoclave.
- ◆ A combination of sulfuric and nitric acids, potassium permanganate, and potassium persulfate. This is the mixture used by the CLP with either a water bath or an autoclave and is identical to the cocktail used with waters.

The available literature and the approved procedures suggest that the combination of acids, permanganate, and persulfate is the most effective in oxidizing organic mercurials.

Until very recently, all authoritative sources required that mercury standards be treated and digested in the same manner as samples. The latest revision to Method 245.1 (May 1994), suitable for water samples, requires that all chemicals added to samples be added to standards, but omits the heating step for standards. Calibration standards are not digested, but laboratory fortified blanks are. The same exemption has not yet been granted for standards associated with solid or semi-solid analyses. If you would still like to digest calibration standards associated with aqueous samples in the same manner as the samples, approved procedures other than 245.1, still allow it.

More rugged automated mercury analyzers are now available. If you use or plan to use one, try to secure from the manufacturer documentation that the analyzer is approved by EPA, or verify that the physical and chemical digestion steps used with the instrument fit into the appropriate allowances described above, and that the reduction to elemental mercury is accomplished quantitatively with stannous sulfate or stannous chloride. For more information, contact Alfredo Sotomayor at (608) 266-9257, or [sotoma@dnr.state.wi.us](mailto:sotoma@dnr.state.wi.us).

## **FOCUS ON ANALYTICAL TECHNIQUES: DRY PURGING**



Dry purging is the term given to a step sometimes incorporated in volatile analyses after purging a sample, but before desorbing the trapped analytes into the analytical GC or GC/MS column. During dry purging, the trap is purged with carrier gas and vented to the atmosphere for a variable amount of time to remove moisture (or methanol) carried over from purging a sample.

The National Exposure Research Laboratory (NERL, formerly EMSL) in Cincinnati advised disallowing dry purging in any drinking water analysis. We had, based on this advice, required laboratories that used dry purging to discontinue the practice. Many laboratories complied readily with this recommendation and noted that once they stopped dry purging, sensitivity generally increased and moisture problems were not appreciably worse after the change.

Since then, proposed changes to SW-846 (pink pages), specifically Method 5030B (Section 7.2.5.1), have incorporated language explicitly allowing dry purging. Partially responding to some of our concerns, Section 7.2.5.1 will be rewritten to add a warning about how to prevent erratic results when using what is now called generically a "moisture removal step". The section, unfortunately, will not give much information on the proper use of the technique.

If moisture carry-over becomes a problem, we recommend solving it preferentially by using a more hydrophobic trap, increasing the bake-out time of the trap in use, or installing a moisture control module on-line between the purge vessel and the trap. Dry purging CANNOT be used for analyzing drinking waters for VOCs. If you must or would like to use dry purging to control moisture problems in other types of samples you should:

- a. Ascertain that the dry purging step is compatible with the trap in use. We know that dry purging is not indicated for silica gel and Tenax traps. It seems to work well for the Vocarb 3000 and 4000 traps.
- b. Limit the dry purging cycle to the minimum time possible.
- c. Assure that the overall sensitivity is not impaired by analyzing a low level standard each day that analyses are performed using dry purging. The standard should contain those VOCs that the Department requires be reported down to their respective MDLs, unless any of them will not be reported on the day of analysis: 1,1,2,2-tetrachloroethane; 1,1,2-trichloroethane; cis-1,3-dichloropropene; trans-1,3-dichloropropene; bromodichloromethane; bromoform; chloroform; chloromethane; methyl-tert-butyl ether (MTBE); methylene chloride; vinyl chloride; dibromochloro-propane (DBCP); and ethylene dibromide (EDB).
- d. Select concentrations above the MDL, but below the LOQ for all the analytes in the low level standard.
- e. DETECT any compound in the low level standard that will be reported on the analysis day prior to analyzing samples.
- f. Take corrective action when any of the analytes in the low level standard are not detected and need to be reported.

These guidelines will allow those laboratories that want to pursue dry purging to demonstrate that using the procedure does not impair sensitivity. For more information contact Alfredo Sotomayor at (608) 266-9257, or [sotoma@dnr.state.wi.us](mailto:sotoma@dnr.state.wi.us).

## ***OIL AND GREASE - HEXANE ISN'T THE ONLY THING THAT'S DIFFERENT!***

As many laboratories have found, when EPA changed the solvent for oil and grease from freon to hexane, they also made significant revisions to the methodology. The method format and requirements are consistent with EPA's efforts toward uniformity. Historically, oil and grease methods did not include quality control. Many laboratories recognized this deficiency and devised mechanisms to monitor method performance, but this suffered from a lack of uniformity, both in selection of material used as a "standard" and interpretation of its results. Method 1664 addresses these concerns by identifying a standard and requiring both sample duplicates and matrix spikes. The implications for sampling are immediately obvious: Persons collecting samples will have to be educated to provide laboratories with a sufficient amount of sample in multiple containers rather than the single bottle required in the past. For laboratories, the implications are far from innocuous, particularly when it comes to interpreting the QC results and taking corrective action following a QC failure. When a matrix spike recovery exceeds control limits, analysts must decide whether the source of the failure is a matrix interference or a sampling problem. Either way, the results may not be reported for compliance monitoring (9.3.4.1). Failing precision control limits creates similar difficulties (9.3.6) discriminating between analytical and sampling variability. We directed these concerns to EPA and are awaiting their response. In the interim, please be aware of the constraints in the method and use a sound decision-making process for corrective action following QC failures.

### ***Possible Solutions For Analytical Problems With HEM***

After repeated attempts to obtain acceptable detection limits, precision, and accuracy, several laboratories questioned whether anyone had been



able to meet method 1664 requirements. The answer is yes, but not without some difficulty. A number of problems have been traced to integrity of the hexadecane/stearic acid spiking solution. Don't overlook the note in paragraph 7.10.1, cautioning that the solution may require warming to completely dissolve stearic acid. Before pipetting the standard, examine it carefully for signs of particulates. When doing the MDL study, take the seven aliquots of the standard quickly. It may be necessary to resuspend the stearic acid even in the brief time it takes to prepare these control samples. We understand that commercial preparations of the hexadecane/stearic acid standard are not immune from similar difficulties. It may help to prepare the standard like other organic standard, placing solid stearic acid into acetone first, then adding the hexadecane and fill to the mark on the volumetric. At least one laboratory reported better success by sonicating the standard before each use. If you use the liquid-liquid extraction and see crystals in your evaporation flasks after driving off the hexane, look at section 11.3.6 and the note that follows it. Excess water in the hexane may result in milky extracts or sodium sulfate crystals in the drying flasks.



### ***Other Tips For Improving Hem Recoveries***

The Laboratory Certification program has been evaluating laboratories for HEM by method 1664 for over one year now, and we have discovered that a few minor adjustments in technique that may help to improve the procedure. First, laboratories that wait for 15 minutes or longer instead of the required 10 minutes for the separation of the hexane and water in the separatory funnel have noticed significantly improved recoveries. Second, a quantitative transfer of extract obtained by carefully rinsing the separatory funnel with a small volume of hexane after the extraction and combining the hexane portions seems to improve recoveries. Any questions about the HEM procedure may be addressed to Donalea Dinsmore, (608) 266-8948, or [dinsmd@dnr.state.wi.us](mailto:dinsmd@dnr.state.wi.us).

## ***REFERENCE SAMPLE NEWS - CHANGES IN THE EPA PROGRAMS***

### ***WS Study No Longer Acceptable For Non-SDWA PAHs***

Unfortunately, the EPA has notified the Laboratory Certification Program that the Water Supply Performance Evaluation Study will no longer contain any Polynuclear Aromatic Hydrocarbons (PAHs) except Benzo(a)pyrene. This means the EPA WS reference samples will no longer be acceptable for WI non-drinking water certification tests such as PAHs by HPLC (in category 13) or PAHs by GC (in category 11). Many laboratories that are certified to test for Polynuclear Aromatic Hydrocarbons by HPLC in Category 13 have historically used the USEPA Water Supply performance evaluation study as their source for these reference samples.

The Safe Drinking Water Certification for PAHs (in category 18) will not be affected by this change. The EPA WS PAH reference sample will be graded using only the Benzo(a)pyrene just as it has been in previous studies.

If you are in search of another reference sample provider for PAHs by HPLC (in category 13), please make sure the new provider uses the appropriate spiking range. If your laboratory has used the WS sample for this test in the past, we urge you to contact either the Wisconsin State Laboratory of Hygiene or one of the other approved reference sample providers (APG, ERA, ASI) and sign up to participate in their program. Be sure the study that you are ordering includes at least four different representative compounds at sufficiently low levels for HPLC work. For fluorescing analytes (all but naphthalene, acenaphthene & acenaphthylene), the true value should be < 1 ppb. For UV absorbing analytes, the true value can be as high as 5 ppb (naphthalene has the highest MDL).



## ***Externalization of P.E.T. Programs***

The EPA has proposed "externalizing" their environmental proficiency testing programs (WP, DMRQA and WS). A notice was published in the July 18 *Federal Register*, pp. 37464-71, and a public meeting was held in August to discuss the options available. The State of Wisconsin did not participate in this meeting, however the State Lab of Hygiene and the DNR have discussed the options and feel that option number two would be the most workable. The Wisconsin DNR and the State Laboratory of Hygiene submitted written comments to the EPA in support of this option. Under Option 2, the EPA would continue to be the standard setting authority for the program, but would work with the National Institute of Science and Technology (NIST) to establish the operational and technical standards used for accrediting sample providers. NIST would be responsible for overseeing compliance with the standards. NIST would also maintain the database and prepare primary standards for distribution to the providers. No decision on which option will be pursued was made at the meeting. We expect that the EPA will announce a decision after sorting through all of the comments received. In the short term, the existing programs will continue to be the primary source of reference samples used in the Wisconsin Laboratory Certification Program. For more information, contact Jeff Ripp at (608) 267-0579, or [rippj@dnr.state.wi.us](mailto:rippj@dnr.state.wi.us)

## ***TRANSFERRING OWNERSHIP & MAINTAINING LABORATORY CERTIFICATION***



1996 has been a year of many changes in the laboratory community. We have seen quite a few mergers, consolidations and name changes that have kept us busy guessing who is who. If your laboratory has or potentially will change hands in the near future, we remind you to contact us as soon as possible and notify us of the change so we can keep our database up to

date and make sure that you continue to receive important mail like certificates and this newsletter.

Chapter NR 149 of the Wisconsin Administrative Code outlines the procedure that must be followed whenever a laboratory changes its name or its ownership.

Maintaining certification through a transfer of ownership or a name change is relatively simple if your laboratory meets certain criteria. Before your laboratory has completed its business transaction, you are required to contact us and help us fill out a phone survey to determine whether or not the certificate can transfer to the new owners. If the certificate can be transferred to the new owner, your laboratory must file an application for transfer of ownership/change of name and pay the revised application fee. If the certificate cannot be transferred, the new owners are required to file an application for certification, and pay the appropriate fees for a new application. If your company is planning to make either an ownership or a name change, please contact John Condrin at (608) 267-2300, or by email at [condrj@dnr.state.wi.us](mailto:condrj@dnr.state.wi.us) for more information.

## ***NEWS & INFORMATION BRIEFS***

### ***Requirements For Ammonia Distillation***



According to ch. NR 219, Wis. Adm. Code, (Analytical Test Methods for Wastewater) all ammonia analyses require distillation unless a comparability study has been done and the study demonstrates there are no interferants present in the effluent. For **municipal wastewater** effluent this study has been done for ion selective electrodes only. You can request this study from our office. The study does not apply to any other methods of analysis or to industrial effluents. Laboratories are required to distill ammonia samples if the laboratory analyzes industrial effluents, or does not use the ion selective electrode to test for ammonia in municipal effluents.



## ***Requirements For Methanol Preservation***

We have received several questions regarding the requirement to preserve volatile organics soil samples in methanol, and would like to clarify that the requirements for methanol preservation in s. NR 700.13, Wis. Adm. Code, (Site Investigations) apply to all volatiles testing in soil, not just for petroleum work in the Leaking Underground Storage Tanks Program.

## ***Nitrate Preservation In Drinking Water***

The Department of Natural Resources has submitted a request, along with supporting data, to EPA Region V, for a variance to drop the requirement to preserve nitrate-nitrogen samples in drinking water with ice. The State Laboratory of Hygiene along with other State Laboratories and EPA Region V generated the supporting data several years ago. The EPA's preliminary response indicates that the variance will not be granted as requested. The DNR will continue to work with the EPA to gain a favorable response. To assist the EPA in approving the variance, a risk-based economic analysis of the preservation requirement is being prepared. For more information, contact Ron Arneson at (608) 264-8949 or [arnesr@dnr.state.wi.us](mailto:arnesr@dnr.state.wi.us).

## ***Reference Samples And Blind Standards Are Not Created Equal***

What is the difference between blind standards and reference samples? Both come from sources outside the laboratory, and ch. NR 149, Wis. Adm. Code, requires laboratories to analyze both types of samples. The requirements for each type of sample are different, and there has been some confusion about what is required for each. A reference sample is a challenge sample whose validated concentration is unknown to everyone in the facility. Reference samples are graded by an approved provider and a final report is sent back to the lab with a grade of pass or fail. Registered and certified laboratories are required to submit

acceptable results from at least one study to the Laboratory Certification Program each year to maintain their certification or registration.

A blind standard is a challenge sample whose validated concentration is known to the lab manager, but not to the analyst who will run the test. For most analytes, ch. NR 149 requires laboratories to analyze three blind standards a year. Since blind standards should be used as internal quality control checks, laboratories must take the appropriate corrective actions and document these actions after failing a blind standard. Laboratories are not required to submit results of blind standard tests to the Department. With problematic tests, laboratories may wish to analyze more than three blind standards a year. For more information about these requirements, contact John Condrón at (608) 267-2300. [condrj@dnr.state.wi.us](mailto:condrj@dnr.state.wi.us).

## ***THE AUDITOR'S CORNER***

Alfredo Sotomayor, Senior Audit Chemist

### ***Confirmation: Accentuate the Positive?***

Some years ago, I came across a reference in *The Economist* ("Tests of the Truth", November 14, 1992) to some wisdom uttered by Sir Karl Popper, who gained fame as a philosopher of science through his *Logic of Scientific Discovery* and other works. The article related Sir Karl's claim that "there is an asymmetry between truth and falsehood; no statements can be proved true, but some statements can be proved false. Science, he continued, is defined by this falsifiability -- it is the fact that they can be proved false, but have not been, which gives accepted scientific statements their value." I had known this, and even remembered that the concept was part of the basis for statistical inference. I thought now on how the idea related to what we loosely call confirmation.

In environmental analyses, confirmations usually follow two paths: a concerted effort to reproduce

a finding, or an attempt to positively identify an analyte. Injecting an already analyzed organic extract into a second column, verifying the presence of an analyte by both an FID and a PID, or simply, re-analyzing a sample on a second day are all examples of efforts to reproduce a finding. Sir Karl would say that these are attempts at proving a finding false. Reproducing the finding fails to prove the finding false. The more times a finding is reproduced, the less likely it is that it will be proved false.

Most mortals take this unlikelihood to mean that the reproduced finding is true, pretty much in the same manner that most of us do not question the existence of a gravitational force when we are awake, even though Sir Karl may conclude that because our daily experiences, which serve as experiments, fail to prove that there is not a gravitational force, Newton's assertion (now a law) has immense scientific value. Because an analyst can **attempt** to prove a positive finding false, analytical results can be invested with more validity than say, asserting the existence of trolls.

In summary, failing to prove something is false is not the same as proving it is true, but if repeated attempts at proving something false fail, we can operate as if the finding were true.

Some analytical chemistry techniques that are based on unique properties of compounds or elements are said to provide positive identification. This is the second path that a confirmation may traverse, and is usually considered more definitive: we have a higher degree of certitude that an analytical result corroborated by a positive identification technique is the identified analyte and not another. Mass spectrometry, atomic absorption and emission spectroscopy provide compound and element specific outputs.

In theory, no two different compounds would have the same mass spectrum and no two different elements would have identical absorption or emission spectra, just like in theory, save for identical twins, no two humans would have the same fingerprints or DNA nucleotide sequence. Fastidious Sir Karl would remark that because the

DNA of all humans has not been compared, proving this exclusive relationship between DNA and a person is impossible; however, when attempts at falsifying this relationship fail frequently, the statement assumes more validity. In environmental analysis we feel compelled to verify positives but are usually content to accept negatives conclusively. Negatives become more definitive as detection limits decrease. Conversely, positives become more questionable as detection limits decrease, but the two trends are not equalized at a midpoint of certitude. Because absolute cleanliness and sterility are impossible to achieve, the lower the depths we probe, the more likely it is we will find skeletons that will interfere with our determination.

Analysts then ponder whether what they detect is real or the result of background contamination. Additionally, because a threshold of signal is needed to get an instrument's juices going, if this amount cannot be corrected, the risk of mistaking instrument background for legitimate signal increases. This explains the need, at times, to confirm, by replicability, low level results even when they are the output of positive identification techniques.

Confirmation can then be understood as a failed attempt at proving a finding false, not a successful proof that a finding is true. The conclusions we make are in more solid ground when the debunking attempt is a very strong one.

Consulting a psychic friend or a fortune teller about a result are weak attempts, compared to re-analyzing an organic extract by mass spectrometry. The reliability of a conclusion can be tied to the strength and frequency of the debunking attempt, which is what statistics do when they give us margins of error and confidence limits.

Rarely is analytical information an end in itself: data are used to substantiate or reach conclusions that go beyond a number, beyond a presence, or an absence. This gets us into the realm of interpretation. To come down from the ethers of

theory consider this cold shower of reality from the same article in *The Economist*:

"Few experimenters plan their work in terms of falsification of particular statements -- though it is possible, after the fact, to say that is what they have been doing. Experiments are often taken as

confirmations of truth, not failures to falsify. And the question of whether an experiment is a falsification, or a confirmation, or indeed has any meaning at all, is not always simply determined. Interpreting experiments is hard and troublesome work."

Hard and troublesome work, indeed! In my next column I will continue this discussion on confirmation.

## FACILITY REQUIREMENTS FOR LANDSPREADING SLUDGE

Laboratories analyzing sludge samples for facilities which landspread the sludge should be aware of the numerical metals ceiling concentration requirements in the Code of Federal Regulations Part 503 and Ch. 204, Wis. Adm. Code. Under these rules, facilities are not allowed to landspread sludge if ceiling concentrations for nine metals are not met. These limits were set by the EPA when the CFR Part 503 regulations were being developed, and are based on protecting health and the environment from reasonably-anticipated adverse impacts. Laboratories testing these types of samples should be aware that any exceedance of these numerical limits may generate a phone call from the facility asking the lab to verify dilutions and calculations to determine the dry weight concentration.

Help the facilities out as much as you can, and remember that communication is very important. The table below lists the metals of concern and their ceiling concentration limits:

NR 204: Table 1

<u>Metal (mg/kg)<sup>1</sup></u>	<u>Ceiling Conc.</u>
Arsenic	75
Cadmium	85
Copper	4300
Lead	840
Mercury	57
Molybdenum	75
Nickel	420
Selenium	100
Zinc	7500

<sup>1</sup>parts per million, dry weight

## ERRATA - YELLOW BOOK CORRECTIONS

The latest revision of the *Laboratory Certification Program Requirements and Guide*, PUBL TS-007-96, (the "Yellow Book") was distributed this Spring to all certified and registered laboratories. Since its distribution, several errors in the text have been discovered. Please make a note of the following corrections in your copy.

**Page ii (inside cover)** - The Regional contact for the Northern Region should be Susan Scobell-Watson, Rhinelander Office, (715) 365-8945, Certification Officer (Municipal). Please remove Tom Atchison's name from the list. The

Regional contact for the Southern District should be Brenda Howald (608) 275-3467. Please Remove Roy Lembcke's name from the list. Also, Laura Forst is the contact person in the Department of Agriculture, Trade and Consumer Protection for microbiological testing in water and milk certification. Her phone number is (414) 923-9607. Michael Kvitrud's phone number (Madison-Central Office) should be changed to (608) 261-8459.

**Page 3-1, Table 3.1** - The EPA WS column should read "N" instead of "Y" for PAHs by HPLC and PAHs by GC.

**Page 3-1, Footnote P** - Should read "may be available".

**Page 4-2, Table 4.2** - PVOC's in sand/soil should have a Required MDL of 25 µg/kg and Target Spiking Level of 25-50 µg/kg.

**Page 13-14, Table F** - The cyanide preservation requirement is incorrect and should read Cool 4°C, NaOH to pH>12. This requirement is consistent with the federal regulations at 40 CFR 141.

### **REMINDERS**

- ✓ Please put your nine digit Facility Identification (FID) number on all correspondence with the Laboratory Certification Program. This insures proper filing and easy identification.
- ✓ The direct FAX line for the Laboratory Certification Program is (608) 267-5231. Please use this when sending transmittals pertaining to certification or registration.
- ✓ The Laboratory Certification Program's World Wide Web home page is located at: <http://www.dnr.state.wi.us/eq/lc/>

### ***Useful Numbers***

Methods Information Communication Exchange (MICE Hotline): (703) 821-4789 (for SW-846 Questions)

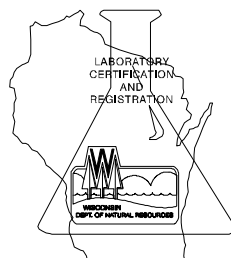
National Technical Information Service (NTIS): (800) 553-6847

Wisconsin DNR - Laboratory Certification (John R. Sullivan, Chief): (608) 267-9753

Wisconsin DATCP- Ag. Cleanup (Duane Klein, Chief): (608) 244-4519

Wisconsin DATCP- Micro Certification (Laura Forst, Certification Officer): (414) 923-9607

Wisconsin DOA Document Sales: (608) 266-3358



### **LABNOTES - FALL 1996**

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